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Analysis and Modelling of an Industrial Pressure Filtration using Process Data^{*}

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Abstract: In order to understand a series of pressure leaf filters located in the downstream line of a bio-based production site, historical process data have been analysed. In general, changing raw materials induce variability into the pressure profiles and thereby cycle durations of the manually reinitialised dead-end filtrations. The absence of a true steady state results in uncertainty about the optimal way of running the filters, and staff members alter the operational specifications frequently. It appears that, in some cases, this propagates disturbances rather than ameliorate them. Statistical analyses are carried out to illustrate the current situation and especially allow quantifying the extent of the uncertainties. Furthermore, significant correlations between process variables are revealed and economically motivated operational objectives are identified. Secondly, working towards on-line predictions of filtration performance, a model is presented. It is based on classical filtration theory and requires only commonly available measurements (pressure, flow, viscosity). The generated predictions are found to be acceptable for many cycles, but in some cases fail due to non-modelled effects, motivating further work.

Keywords: Biosystems and bioprocesses; Downstream processing; Parameter and state estimation; Data mining tools; Modelling and identification; Pressure leaf filtration

1. INTRODUCTION

To date, cake filtration is one of the workhorses of the biochemical industry when it comes to the separation of demanding slurries with a high concentration of suspended solids. Due to its versatile applicability and robust separation properties, both of which have been demonstrated over many decades, it is an established technology and far from being replaced in the foreseeable future. A trend toward automation in industrial filtration is not new - the economic success of automatically discharging horizontal leaf filters was shown already in the 1970s, see Rushton et al. (1996). Especially when handling pharmaceuticals or food products, closed systems are desirable for the reduced risk of contamination. Nevertheless, many current plants have downstream processes where manual operation (also on decision-making level) is regarded normal. There are various reasons for this, but in particular persistently changing process conditions are challenging from a control engineering perspective. They can generally be attributed to short-lived product cycles and inconsistent feedstock (weather, region, pre-treatment, etc.). The necessary adaptivity for dealing with these uncertain conditions is provided by keeping operators in the loop and would otherwise require sophisticated automation solutions. In general, filtration, a seemingly simple processing step,

is non-trivial from an operational point of view. Discrete events such as cleaning (the removal of filter cake) and re-initialisation happen on a frequent but irregular basis. By nature, these disruptions upset the steady state of the surrounding line. Buffer tanks have a mitigating effect, but in reality one still finds propagation through the system, which is why intermittent filtration is prone to induce variability into process and ultimately product.

In many biochemical plants, the absence of a true steady state is an obstacle in the quantitative analysis of downstream processes. Thus, in order to provide a working basis, it is necessary to reduce uncertainties to a feasible minimum. Currently the incentive for vast capital investments in downstream processes is limited, as competitiveness predominantly originates from a high degree of product-innovation. Beyond this, the long-term ambition of transitioning from recipe-driven open-loop production to *lean*, automated plants requires sophisticated change management. Undoubtedly a challenging endeavour, and it is for industrial practitioners and academia to work together, recognising and exploiting progress that has been made in sensor technology, monitoring- and ultimately methodological tools.

Production processes that have been running for decades have seen changes for the better due to the involvement of capable engineers and operators. However, these heuristic findings, while robust in their applicability, are often restricted to a local *modus operandi*. A first step toward gaining a deeper understanding as well as applying quantitative analyses to any process is the derivation of a reliable

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process model. Classical filtration theory has its pitfalls when it comes to modelling and ultimately designing industrial equipment, as delineated early by Ruth (1946). Likewise, some of the more recent modelling approaches are hard to match with industrial practice, where filter cake properties change frequently and cannot be determined in recurring laboratory experiments or by utilising exhaustive sensor technology due to the involved expenses. This work encompasses a description of the complex operational aspects that are inherent to intermittent filtration processes at hand of a comprehensible case study. Statistical analyses of industrial process data are carried out, important correlations identified and the results thoughtfully visualised. A predictive model for industrial scale filtration using available process data only is presented. It is largely based on classical filtration theory, and a number of forecasts are found to miss, likely due to non-modelled effects, motivating further work in this area.

2. FILTRATION THEORY

Scientific approaches to understanding filtration date back to the 1850's, when Darcy noted the proportional relation between pressure drop Δp and liquid flow Q through a packed bed of solids (1). Physically arguing, the solid-liquid suspension passes through channels that form in the bed. Friction losses, depending on viscosity and flow regime, occur when the medium passes over a surface. Larger particles will start to accumulate on the upstream side of the cake, which therefore grows over time. Smaller particles gradually adhere to walls and cavities while passing through the channel-matrix. Pressure losses across the cake are thus linked to the depth of the permeated media, which furthermore influences the quality of a separation. However, characteristics such as particle size distribution, shape and morphology, surface charge, and pH will determine the pressure gradient throughout the bed (Tarleton and Willmer (1997)). It is convenient to express the flow-pressure drop relation by means of a cake resistance α :

$$Q = \frac{A \Delta p}{\mu L \alpha} \quad (1)$$

Here, L denotes the measure for the depth of the bed, A the cross-sectional area and μ viscosity. In reality α is not constant throughout the cake, as nearly all filter cakes exhibit compressibility - meaning that permeability properties in the cake are not constant. For a limited range of pressures, an empirical approximation is given by

$$\alpha(\Delta p, n) = \alpha_0 \Delta p^n, \quad (2)$$

where n is a compressibility index (0 denoting a non-compressible material) and α_0 a reference resistance. If a more convenient expression is desired, one finds a remedy in averaging over the cake depth, as documented e.g. in Tien (2006), which then yields

$$\bar{\alpha} = (1 - n) \alpha_0 \Delta p^n. \quad (3)$$

In order to extend the descriptiveness of equation (1) to an entire filtration cycle, the relationship between cake growth and time must be established. If the concentration of accumulating particles on the top-layer (\bar{c}) and likewise the cake's structural integrity are sufficiently constant,

$$L = \frac{\bar{c} \int Q dt}{A} = \frac{\bar{c} V}{A} \quad (4)$$

yields the desired equivalence, in which V denotes the amount of processed filtrate. Thus, equation (1) with (4) and ultimately (3) yield the necessary link between time of filtration, processed filtrate, and pressure drop:

$$\frac{dt}{dV} = \bar{\alpha} \mu \bar{c} \frac{V}{A^2 \Delta p} + \frac{\mu R_m}{A \Delta p} \quad (5)$$

In (5), a second term has been included to account for the medium resistance across the membrane (R_m). It is a common assumption to view R_m as constant, furthermore is the cake resistance typically larger by several orders of magnitude. Reliable first principle models are scarce and complex, thus it should be regarded best practice to determine any α , c or R_m experimentally in lab- or ideally pilot scale experiments. However, one should keep in mind that even then, cross-scale validity is usually an issue, as discussed in Tarleton and Hancock (1997).

3. FILTRATION PRACTICE

Filtration units deployed in continuous production lines are often controlled such that they yield a uniform filtrate flow (constant rate filtration) throughout a cycle. Ideally, a cycle is meant to come to an end when the maximum operating pressure p_{max} of either pump or membrane is reached. Equipment is designed such that large surface areas can be accommodated small pressurised vessels, normally by stacking many plates side-by-side, similar to the layout of plate heat exchangers. The membranes alone provide a weak separation, as a consequence high flow rates can lead to penetration of the septum in the early stages of a filtration. This risk is commonly reduced by applying a precoat layer, using an inert material. Throughout a filtration the cake then grows on the membrane surface, and each two plates ('leaves') are arranged such that they form a cavity through which the filtrate is discharged. In the case of highly compressible solids, filter aid can be added during the filtrations as a body feed. This has a positive effect on porosity, enhancing capacity and longevity of the filter cycles. A rather detailed study on the effect of filter aid in a bioprocess is presented by Meindersma et al. (1997).

3.1 Operational Aspects

In the case of manually cleaned filters, personnel expenses contribute a large part of the overall operational costs, which has implications for the optimal utilisation of the equipment. Two not mutually exclusive but dependent objectives, subject to a soft constraint, could be identified:

- Operate the units such that the number of total manual filter cleanings n is minimised (objective)
- Use manpower efficiently by maximising the amount of cleaned filters per operator and shift (objective)
- Process all upstream feed as steadily as possible by making use of the buffer capacities (constraint)

Constraint: Firstly, in order to maintain plant capacity, it is necessary that

$$\bar{Q} = \sum \eta_i \bar{Q}_i = Q_{plant}, \quad (6)$$

where \bar{Q} is the average flow coming from all filters and $\bar{Q}_i = \int Q_i(t) dt \cdot T_{fi}^{-1}$ describes the average flow per cycle on a single filter. See (7) for a derivation of the equipment

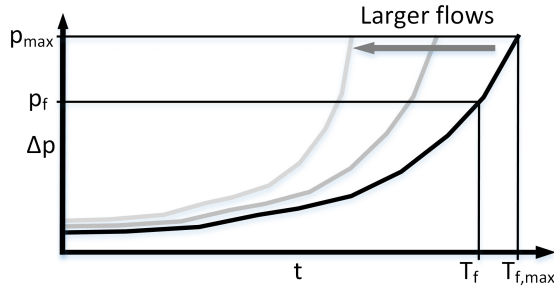


Fig. 1. Relation between flow rate, time, and final pressure efficiency measure η_i . Buffer tanks are indispensable due to the intermittent nature of the process and allow temporarily altering flow rates without upsetting the surrounding line. Thus, as long as tank capacity suffices, equation (6) can be expressed as a soft constraint in averaged values, which ideally allows scheduling the end times by manipulation of flow rates. In the case study plant this is carried out by the operators on a rule-of-thumb basis.

Objective 1: The number of necessary manual actions is minimised if the equipment efficiency measure for each filter i , namely

$$\eta_i = \frac{T_{f_i}}{T_{f_i} + T_{cl}} \quad , \quad T_{f_i} = f(Q, \Delta p, Z) \quad , \quad (7)$$

is maximised. Cleaning and reinitialising a filter is a standardised procedure and takes a certain time T_{cl} . The varying filter cycle durations are denoted by T_{f_i} . Lastly, Z indicates disturbances such as e.g. fluid properties. To maximise η_i , one must assure that the maximum amount of retentate (solids) is accumulated on the membranes before being removed. Intuitively this suggests striving for an even distribution of the workload between units; a data-driven, quantitative relation between average filtrate flow and cumulative amount of filtrate per filtration could not yet be derived with sufficient reliability. Furthermore, flow - and thus pressure - increases which could cause cake blinding should be avoided. Lastly, a filter is cleaned more often than necessary if $p_f < p_{max}$ on take out, as this leads to a decrease in T_f (figure 1) and thereby η .

Objective 2: The operator load factor can be expressed as

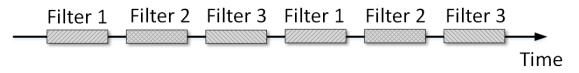
$$\xi_{operator} = \frac{N T_{cl}}{T_{shift}} \quad , \quad (8)$$

where N denotes the number of manual actions. Thus, an ideal filtration area would at all times be run such that operators do not experience idle times between consecutive filter cleanings (figure 2a). In reality, long cycles in combination with the flow rate constraint must result in idle time, unless the plant can handle a flow rate increase. However, scheduling uncertainty is so large that non-productive operator time (figure 2b) can emerge (or units are taken out prematurely) even in cases where plant throughput cannot be maintained. This has strong economic implications due to the involved production losses and calls for a new, robust operational paradigm.

3.2 Pressure Leaf Filtration - Case Study Setup

The filtration unit under study is located within one of the pectin production lines operated by CP Kelco ApS in Denmark. *Pectin* is a gelling and thickening agent used pre-

a: Ideal – operator without non-productive time



b: Uncertainty leads to non-productive time

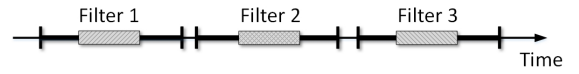


Fig. 2. Operator engagement in reinitialisations (grey bars)

dominantly in the food industry, and is generally leached from citrus peels in acidic extractions. Intuitively, one would expect a gelling agent to be viscous in solution, and it is indeed a challenging task to separate the extraction broth. A multi-step filtration is necessary, and initially, first- and second stage solids are removed. Subsequently the suspension is fed to the pressure filtration area, where several manual and automatic units run in parallel. As the manually cleaned filters are of a greater interest from a process-optimisation point of view, the automatic ones have been neglected in the analyses for now. All filters are of identical design, however, two of the five have a smaller membrane area. Precoating is necessary and filter aid is supplied as a body feed throughout the filtrations. Notably, the precoat layer is applied using process fluid, thus one should expect some inconsistencies in its composition. The operators are instructed to terminate a filtration upon reaching a pressure threshold. The vessel is then opened, leafs and supporting rack are slid out, and finally the membranes are hosed down with water in order to remove the filter cake. Thereafter, the vessel is reinitialised, which includes running in precoat-recycle until all filter aid is evenly distributed on the membranes. In this procedure, it is up to the operators to schedule the filtrations such that no blockages arise. Due to the amount of information (multiple filters) and the non-linearity of the pressure profiles, this is a complex task, and the quality of these decisions is sometimes questionable. Pressure filtration can become the bottlenecking step, and it is unclear whether this is due to disturbances alone or if suboptimal operation is an issue. Thus, in order to prevent the operators from involuntarily propagating disturbances or scheduling in an erroneous way, the desire for a more deterministic operating regime with augmented decision-making has been expressed.

4. STATISTICAL ANALYSIS

Before attempting to model the filters, historical process data have been analysed to gain an overview of the modelling scenario, furthermore to investigate uncertainties and correlations. Operational data from some of the pressure leaf filters, logged at 1-minute intervals over the course of a month, have been analysed. Due to the high number of regarded cycles (>250), manual processing of the pressure profiles was not feasible and an algorithm has been developed to extract them from the time-series data.

4.1 Data Handling

The data are logged on a continuous time axis, and the individual filtration cycles have been identified with an algorithm that recognises the initiation of a new run

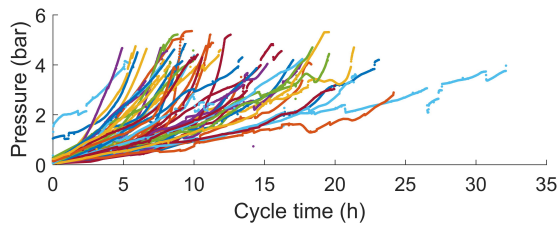


Fig. 3. Pressure profiles on cycle time axes

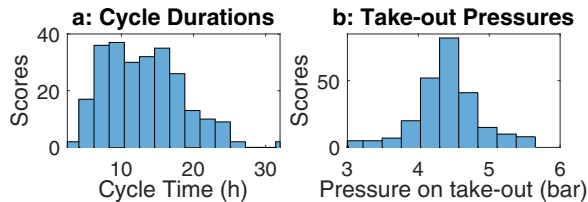


Fig. 4. Distribution of cycle times and take-out pressures based on the pressure profile. A new cycle is generally characterised by a sudden decrease in pressure below a (variable) threshold:

$$p_{i-1} - p_i \geq \epsilon_1 \quad | \quad p_i \leq \epsilon_2, \quad i = 1, \dots, \text{end} \quad (9)$$

All cycles could be uniquely identified with $\epsilon_1 = 1 \text{ bar}$ and $\epsilon_2 = 0.5 \text{ bar}$. However, it was necessary to eliminate data-points between cycles, which can occur if data is being logged even though a filtration has come to an end. Checking for

$$p_{i-1} - p_{i+1} \geq \epsilon_3 \quad | \quad \frac{p_i - p_{i+1}}{p_{i-1} - p_i} \geq \epsilon_4, \quad i = 1, \dots, \text{end}, \quad (10)$$

with $\epsilon_3 = 2 \text{ bar}$ and $\epsilon_4 = 0.01$ allowed to remove all outliers of this type. The check against ϵ_4 identifies whether the gradient undergoes a change in sign during the transition, another necessary condition for a new cycle.

4.2 Frequentist Analysis

Looking at figure 3, where the pressure profiles have been stacked on their cycle time axes, the uncertainty becomes apparent. There is some variability in the final pressures, but first and foremost the cycle durations catch the eye, as they stretch from few hours to more than a day. This is put into a different representation in figure 4a, where one can see the majority of cycles coming to an end between 8 and 18 hours with a tail spanning much further. Figure 4b shows a histogram of the final pressures. The exhibited profile - unlike the previous one - seems to somewhat resemble a normal distribution. This could be explained with the randomness induced by the operators when they decide to take a filter out of production. Notably, the mean of this distribution lies below the pressure threshold that is specified in the operational guidelines.

4.3 Correlated Process Variables

It is interesting to determine whether there are distinct differences between the way each filter is operated and, ultimately, performs. A first visual analysis can be carried out at hand of figures 5a and b. There is some uncertainty in the calculation of the points in time when a cycle is

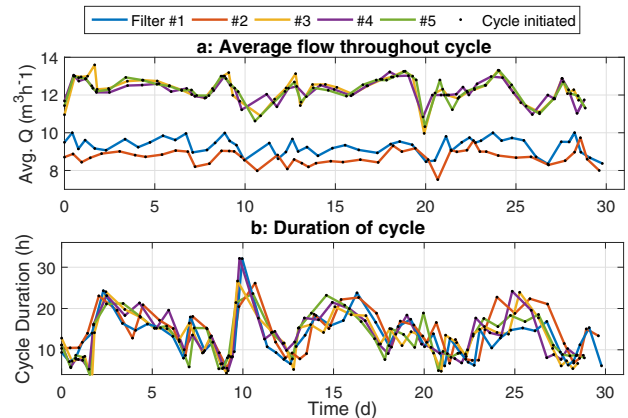


Fig. 5. (Scaled) average flows and actual durations

initiated, but an unquestionable correlation between the average flows and durations of the cycles across all filters can be determined. From an operational point of view, similar flow profiles are to be regarded as positive due to the even distribution of the workload. However, it is interesting to note that the scaled but representative average flow rates vary significantly with time. Furthermore, flow rates are generally lower on filters 1 and 2 due to their smaller membrane area, but it is surprising to see that there is a distinct difference between the two, as they are equal-sized. Upon inquiring, the operating crew stated that Filter 2 is believed to perform worse than Filter 1. However, looking at the averaged values, given as [mean (st. deviation)] in table 1, the differences in cycle times and ultimately the number cycles per month should be noted. Durations on Filter 2 are significantly higher, thus it is likely that the operators run the filter at lower flow rates out of an erroneous assumption. Looking at the total filtrate output per month, Filter 2 performs only at about 93% of Filter 1. This is a good example of hidden capacity that can be revealed by systematically analysing process data. A different approach at visualising the inter-filter correlations is shown in figure 6. The centres of the black circles denote a point in time at which a cycle has been started, and it is obvious that periods of short or long filtrations are experienced across the filters. The coloured lines are normalised and scaled representations of the average flow rates of these cycles. One can see that higher flow rates imply shorter cycles, however, looking closely, there are exceptions to this rule. Lastly, a normalised and scaled viscosity measure has been added. It is plotted on the line corresponding to Filter 4 (where the measurement is taken). However, one should expect the fluid properties on the filters to be similar, as they are supplied from the same tank. It is evident that, even though there is a notable correlation between viscosity and filtration performance, there are exceptions to this rule also. Summarising, figure 6 shows how filtration theory and industrial practice do not

Table 1. Cycle statistics across filters

Filter	Flow (m^3h^{-1})	Duration (h)	Nr. of cycles
#1	9.3 (0.44)	12.6 (5.1)	53
#2	8.7 (0.41)	14.1 (5.5)	48
#3	12.3 (0.69)	12.8 (5.4)	50
#4	12.2 (0.55)	13 (5.7)	50
#5	12.2 (0.65)	13 (5.3)	50

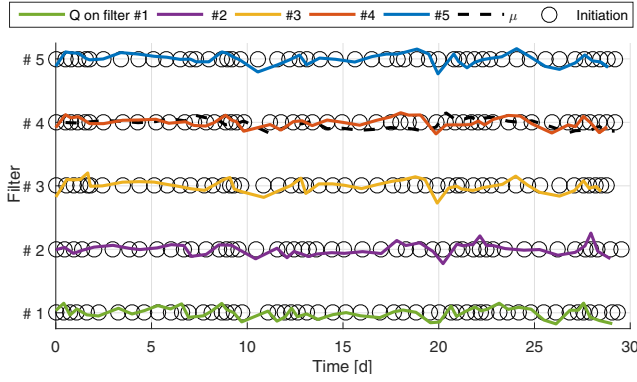


Fig. 6. Clustering of short cycles & viscosity effects

align at all times. Due to the equal number of total cycles (table 1), the correlation matrix $\Sigma_{\sigma_{ij}}$ between the cycle durations on the large filters can be calculated with ease. Beyond this, there is a correlation between the durations of consecutive cycles on each filter ($\sigma_{k,k+1,i}$), but it is weaker and applies to a lag of one cycle only. Remarkably, the correlation is quite low on Filter 1. This could be related to the fact that operators choose to adjust the flow rate such that it is slightly higher than that on Filter 2 (which had been classified as a problematic filter by the operators).

$$\Sigma_{\sigma_{ij}} = \begin{pmatrix} 1 & 0.77 & 0.78 \\ 0.77 & 1 & 0.76 \\ 0.78 & 0.76 & 1 \end{pmatrix}, i, j = 3, 4, 5$$

$$\sigma_{k,k+1,i} = (0.22, 0.43, 0.42, 0.36, 0.48)^T, \forall i, k$$

5. MODELLING THE FILTRATION CYCLES

The general ambition of this section is to assess whether it is possible to derive a predictive filtration model using only process data. In this case, due to the high variability between the cycles, the model is fitted independently for each cycle instead of looking for an optimal parameter set across cycles, or even filters. With $dV/dt = Q$ and (3), the filtration equation - foregoing the membrane resistance and solved for the pressure difference - reads

$$\Delta p = \left(Q \frac{V \bar{\alpha}_c \mu}{A^2} (n-1) \right)^{\frac{1}{1-n}}. \quad (11)$$

Cake resistance and compressibility index are to be fitted. Lacking the necessary data, the solids concentration \bar{c} cannot be uniquely determined, thus the lumped parameter $\bar{\alpha}_c = \bar{\alpha} \bar{c}$ is estimated. Beyond this, flow rates fluctuate notably in a number of cycles. Thus, in order to reduce the model error, the integral for V has been solved using trapezoidal integration rather than assuming $V = Qt$ (constant rate filtration). For all affected cycles, this has improved the quality of the fit. However, in order to predict performance, it needs to be a strict guideline that flow rates remain unchanged unless absolutely necessary, as changing flow rates, especially if not based on deterministic decisions, rapidly deteriorate the precision of the predictions. Instead of including a term for the membrane resistance, the pressure profiles have been truncated by an initial offset - which is later-on re-added to the model output. This has two major implications:

- (1) The number of variables to be estimated is reduced. This is positive, as high correlations imply low pre-

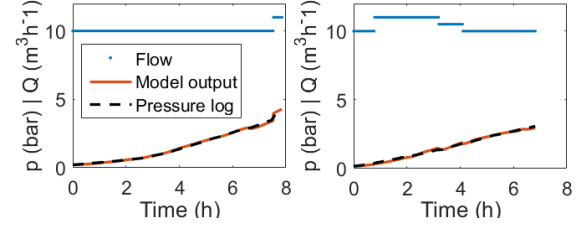


Fig. 7. Cycles with and without changing flow rates

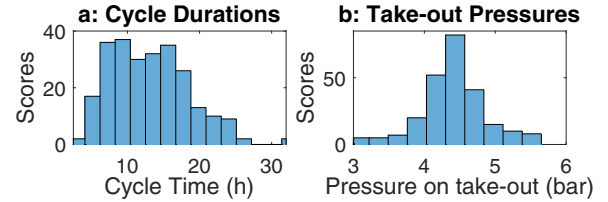


Fig. 8. Distribution of fitted parameters

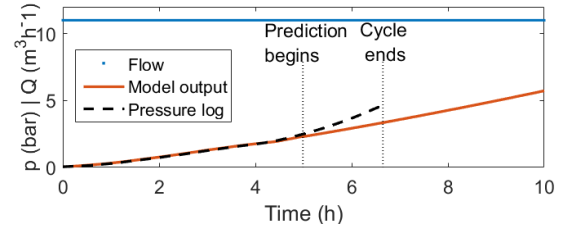


Fig. 9. Prediction misses trend despite of smooth profile

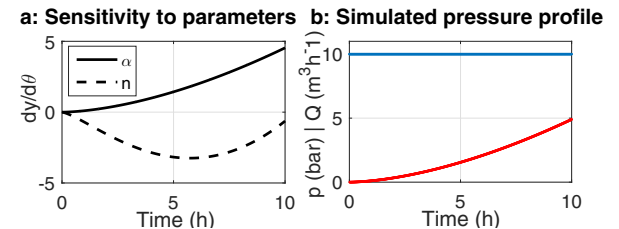


Fig. 10. Local sensitivity measure for $\alpha = 2, n = 0.4$

dictability, and are expected due to model structure and the limited amount of data available for fitting.

- (2) It is a parameter that can be iteratively adjusted without upsetting the model outcome much.

Looking at figure 7, the chosen model seems reasonable. Despite of minor upsets to the flow (right-hand side), the model fit is within acceptable bounds. For cycles with extreme fluctuations in the flow profiles, the model fit will generally be worse, but this is not strictly applicable for all cases (see figure 12a, where a good model fit can be achieved despite of an unsteady profile). Beyond this, the fitted parameters are often highly correlated, for the two depicted cycles at around 0.9. Looking at all cycles, this correlation is seen to move between 0.3 and 0.99. Consequently one must deduce that this approach is likely not able to yield a reliable prediction. The spread of the fitted parameters (all cycles on one filter for the entire month) is visualised in figure 8.

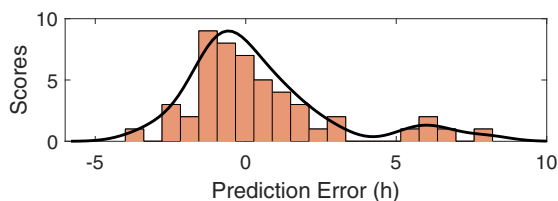


Fig. 11. Prediction errors, 75% of cycle, $n = 0.45$

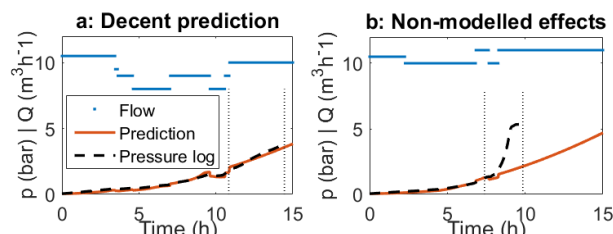


Fig. 12. a: Example of a good prediction, b: Example of failure due to non-modelled effects

5.1 Predictivity of the Model

To assess predictivity, the parameters are estimated at a certain point before the end of a cycle. The predicted model outcome is then compared with the actual values. Under the premise of a flow guideline for the operators, knowledge of the flow profile over the prediction horizon has been assumed. The viscosity is set to the mean value of the past 60 minutes before the prediction begins. In figure 9, the outcome is based on knowledge of 75% of the profile of the actual cycle. This cycle was not chosen deliberately. It has been selected for its steady flow profile, which implies a small modelling error. In spite of this, the prediction misses the compressibility-induced steep pressure increase toward the end completely. This is seen throughout all cycles, and looking at the unlike signs in the local sensitivity function (Sin and Gernaey (2016)), based on the representative parameter values $\alpha = 2, n = 0.4$ in figure 10, one must conclude that difficulties are to be expected in the simultaneous estimation of both parameters. Thus, with figure 8b in mind, the compressibility index is fixed at its maximum likelihood value. However, in practice the predictions have shown to be better when n assumes a slightly higher value. Ultimately, it has been iteratively adjusted by looking at the prediction errors across all cycles (restricted to one filter). The error distribution for $n = 0.45$ and knowledge of 75% of the cycle profile is plotted in figure 11. For most of these predictions, the error is seen to be bounded by approximately $\pm 2h$. Figure 12a shows that good guesses are possible also when the flow rate is unsteady. However, it is to be expected that the quality of the predictions would improve significantly under an operating regime with constant / deterministically adjusted flow rates due to the minimisation of non-modelled effects such as cake blinding and particle rearrangement. From figures 12b and 13 it can be concluded that the algorithm, while performing rather well for a large number of cycles, is not robust enough for rigorous scheduling. Furthermore, it can be learned that there is only a weak correlation (0.2) between absolute error and duration of a cycle.

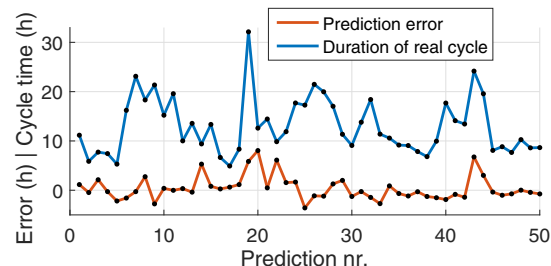


Fig. 13. Correlation between pred. error and cycle duration

6. CONCLUSION

One should note the complexity arising in industrial-scale filtrations due to the nature of the process as well as the extent of regularly occurring disturbances. The situation has been extensively elucidated and furthermore predicated upon real process data. A predictive model poses a first step toward a new operational regime that relies less strongly on operator expertise, thereby reducing the risk of human failure. It is found that the uncertain circumstances and a series of irregular events cannot be entirely captured by the model. This deteriorates the outcome of the predictions, but it is unlikely that the quality of the guesses could be substantially improved by the use of a more sophisticated model or wet lab experiments as many uncertainties are believed to arise from operational disturbances such as e.g. properties of the precoat layer. On the other hand, correlations between consecutive cycles as well as the interdependencies between the parallel units should be acknowledged. They suggest an inferential superstructure that weighs filtration theory based predictions and statistically motivated values against each other. If this proves to enhance robustness sufficiently, visualised predictions or even a scheduling algorithm can be envisioned.

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